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(54) ACTIVE MATERIAL AND POSITIVE PLATE FOR NONAQUEOUS ELECTROLYTE SECONDARY BATTERY, AND THE NONAQUEOUS ELECTROLYTE SECONDARY BATTERY

(57)Abstract:

PROBLEM TO BE SOLVED: To improve the characteristics, especially the storage characteristic of a secondary battery using a nonaqueous electrolyte.

SOLUTION: A lithium nickel composite oxide used as an active material for a nonaqueous electrolyte battery is represented by the chemical formula, $LixNi_yM_{1-y}O_2$ { x : 1. $10 \geq x \geq 0.98$, M represents at least one or more from among Co, Mn, Cr, Fe, Mg, Al, y : $0.95 \geq y \geq 0.7$ }, a globular or elliptical particles formed by aggregation of primary particles having a particle size of $2 \mu m$ or less, and has specified physical properties.

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CLAIMS

[Claim(s)]

[Claim 1] Chemical formula $\text{Li}_{x}\text{Ni}_{y}\text{M}_{1-y}\text{O}_2$ (x:1.10>=x>=0.98 and M are any one or more kinds of Co, Mn, Cr, Fe, Mg, and aluminum, and y:0.95>=y>=0.7), and primary particles 2 micrometers or less gathered and which consists of spherical or a lithium compound nickel oxide which is an ellipse ball-like particle and is equipped with the following physical properties.

1) the BET specific surface area by which the whole space product to which the spatial body product which has the pore radius of 30 or less A has a pore radius not more than less than [10%] 230A to the whole space volume is measured by below 0.002cm³/g³ nitrogen gas adsorption -- 0.15-0.3m²/g⁴ mean particle diameter -- 10-16-micrometer⁵ tap density -- the spatial body product of 2.0 - 3.0 g/cm³ pore -- 0.0015-0.06cm³/g -- [Claim 2] Said lithium compound nickel oxide is lithium salt and chemical formula $\text{Ni}_{y}\text{M}_{1-y}(\text{OH})_2$ (it Co(es) M). Any one or more kinds of Mn, Cr, Fe, Mg, and aluminum and the different-species element dissolution nickel hydroxide expressed with y:0.95>=y>=0.7 are mixed. The active material for nonaqueous electrolyte rechargeable batteries which consists of a lithium compound nickel oxide according to claim 1 characterized by being the object obtained by grinding the massive object obtained by calcinating in a 700-900-degree C temperature requirement, and classifying.

[Claim 3] In the monotonous and constituted positive-electrode plate for nonaqueous electrolyte cells which uses a lithium compound nickel oxide as a principal component, supports the electric conduction material which consists of carbon material, a binder, and said lithium compound nickel oxide, and gives conductivity Chemical formula $\text{Li}_{x}\text{Ni}_{y}\text{M}_{1-y}\text{O}_2$ (it Co(es) x:1.10>=x>=0.98 and M) The positive-electrode plate for nonaqueous electrolyte rechargeable batteries with which it was expressed with any one or more kinds of Mn, Cr, Fe, Mg, and aluminum, and y:0.95>=y>=0.7, and primary particles 2 micrometers or less gathered and which consists of spherical or a lithium compound nickel oxide which is an ellipse ball-like particle and is equipped with the following physical properties.

1) the BET specific surface area by which the whole space product to which the spatial body product which has the pore radius of 30 or less A has a pore radius not more than less than [10%] 230A to the whole space volume is measured by below 0.002cm³/g³ nitrogen gas adsorption -- 0.15-0.3m²/g⁴ mean particle diameter -- 10-16-micrometer⁵ tap density -- the spatial body product of 2.0 g/cm³ - 3.0 g/cm³ pore -- 0.0015-0.06cm³/g -- [Claim 4] a positive-electrode plate -- a weight ratio -- lithium compound nickel oxide: -- electric conduction material: -- the positive-electrode plate for nonaqueous electrolyte rechargeable batteries according to claim 3 characterized by a binder being 100:1-10:2-8.

[Claim 5] said plate -- more than aluminum 95% -- the positive-electrode plate for nonaqueous electrolyte rechargeable batteries according to claim 3 which is the foil to contain.

[Claim 6] A lithium compound nickel oxide is lithium salt and chemical formula $\text{Ni}_{y}\text{M}_{1-y}(\text{OH})_2$ (it Co(es) M). Any one or more kinds of Mn, Cr, Fe, Mg, and aluminum and the different-species element dissolution nickel hydroxide expressed with y:0.95>=y>=0.7 are mixed. The positive-electrode plate for nonaqueous electrolyte rechargeable batteries according to claim 3 characterized by being the object obtained by grinding the massive object obtained by calcinating in a 700-900-degree C temperature requirement, and classifying.

[Claim 7] The positive-electrode plate for nonaqueous electrolyte rechargeable batteries according to claim 3 characterized by the lithium compound nickel oxide eating into said plate.

[Claim 8] The positive electrode which uses a lithium compound nickel oxide as a principal component, and the negative electrode which makes a subject electrochemically the carbon material or oxide of a lithium in which occlusion release reaction is possible, In the nonaqueous electrolyte cell which serves as the organic electrolytic

solution, and a separator and the case which inserts these generation-of-electrical-energy elements from the obturation plate equipped with the relief valve before initial charge and discharge said lithium compound nickel oxide Chemical formula $\text{Li}_x\text{Ni}_y\text{M}_1-y\text{O}_2$ (it Co(es) $x:1.10>=x>=0.98$ and M) The lithium compound nickel oxide with which it was expressed with any one or more kinds of Mn, Cr, Fe, Mg, and aluminum, and $y:0.95>=y>=0.7$, and primary particles 2 micrometers or less gathered and which is the particle of the shape of spherical or an ellipse ball, and consists of the following physical properties, The amount of nonaqueous electrolyte per cell capacity 1Ah is a nonaqueous electrolyte rechargeable battery which is 3/Ah 3.0-6.0cm using the electric conduction material which consists of carbon material, a binder, and the monotonous and constituted positive-electrode plate for nonaqueous electrolyte cells which supports these and gives conductivity.

1) the BET specific surface area by which the whole space product to which the spatial body product which has the pore radius of 30 or less A has a pore radius not more than less than [10%] 230A to the whole space volume is measured by below 0.002cm³/g³ nitrogen gas adsorption -- 0.15-0.3m²/g⁴ mean particle diameter -- 10-16-micrometer⁵ tap density -- the spatial body product of 2.0 g/cm³ - three or more 3.0 g/cm⁶ pore -- 0.0015-0.06cm³/g -- [Claim 9] a positive-electrode plate -- a weight ratio -- lithium compound nickel oxide: -- electric conduction material: -- the nonaqueous electrolyte rechargeable battery according to claim 8 characterized by a binder being 100:1-10:2-8.

[Claim 10] said plate -- more than aluminum 95% -- the nonaqueous electrolyte rechargeable battery according to claim 8 which is the foil to contain.

[Claim 11] A lithium compound nickel oxide is lithium salt and chemical formula $\text{Ni}_y\text{M}_1-y(\text{OH})_2$ (it Co(es) M). Any one or more kinds of Mn, Cr, Fe, Mg, and aluminum and the different-species element dissolution nickel hydroxide expressed with $y:0.95>=y>=0.7$ are mixed. The nonaqueous electrolyte rechargeable battery according to claim 8 characterized by being the object obtained by grinding the massive object obtained by calcinating in a 700-900-degree C temperature requirement, and classifying.

[Claim 12] The nonaqueous electrolyte rechargeable battery according to claim 8 characterized by the lithium compound nickel oxide eating into said plate.

[Claim 13] The solvent of nonaqueous electrolyte is a nonaqueous electrolyte rechargeable battery according to claim 8 characterized by being the mixed solvent which added at least 20% or more of chain-like carbonate, chain-like ester, or both at the rate of a volume ratio to ethylene carbonate or propylene carbonate.

[Claim 14] Said chain-like carbonate is a nonaqueous electrolyte rechargeable battery according to claim 8 characterized by being dimethyl carbonate or ethyl methyl carbonate.

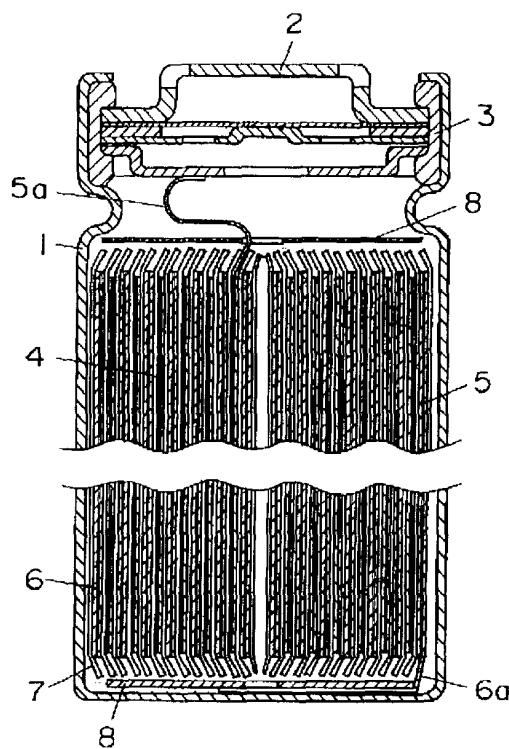
[Claim 15] Said chain-like ester is a nonaqueous electrolyte rechargeable battery according to claim 8 characterized by being methyl propionate.

[Claim 16] A separator is a nonaqueous electrolyte rechargeable battery according to claim 8 which is a fine porosity film made from an organic macromolecule.

[Claim 17] Said separator is a nonaqueous electrolyte rechargeable battery according to claim 16 characterized by consisting of polyethylene, polypropylene, or an object that combined both.

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Drawing selection Representative drawing



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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the cell property improvement especially about a nonaqueous electrolyte rechargeable battery and a positive-electrode plate, and positive active material.

[0002]

[Description of the Prior Art] In recent years, portable-izing of consumer electronics and cordless-ization are progressing rapidly. Although the nickel-cadmium battery or the closed mold small lead accumulator is bearing a role of current and a power source for a drive of these electronic equipment, the request of the formation of a high energy consistency of the rechargeable battery used as the power source for a drive and the formation of small lightweight is strong as portable-izing and cordless-ization are progressed and established. Moreover, it is observed as a power source for cellular phones, and the request to protraction of duration of a call and an improvement of a cycle life is very large with expansion of a rapid commercial scene in recent years.

[0003] The lithium compound transition-metals oxide in which high charge and discharge voltage is shown from such a situation, For example, LiCoO₂ (for example, JP,63-59507,A official report) and LiNiO₂ (for example, U.S. Pat. No. 4302518) which aimed at high capacity further, two or more metallic elements and the multiple oxide (for example, Li_yNi_xCo_{1-x}O₂: JP,63-299056,A, Li_xM_yN_zO₂ (it is a kind at least however, M was chosen from Fe, Co, and nickel --)) of a lithium The nonaqueous electrolyte rechargeable battery with which N was chosen from Ti, Mg, Cr, and Mn and which used kind:JP,43-267053,A for positive active material at least, and used insertion of a lithium ion and balking is proposed.

[0004] Moreover, the improving method about mean particle diameter (JP,1-304664,A, JP,6-243897,A, JP,6-290783,A, JP,7-114942,A) or a configuration (JP,6-267539,A, JP,7-37576,A) is proposed also about the physical properties of positive active material, for example.

[0005]

[Problem(s) to be Solved by the Invention] LiNiO₂ had large polarization at the time of charge, although the high energy consistency was expected compared with LiCoO₂ and development was furthered in every direction, and since the oxidative degradation electrical potential difference of the electrolytic solution was reached before being able to take out Li enough, a large capacity expected was not obtained.

[0006] In order to solve such a problem, what permuted some nickel elements by Co is used for positive active material, and the nonaqueous electrolyte rechargeable battery using insertion of a lithium ion and balking is proposed.

[0007] For example, in JP,62-256371,A, the lithium compound nickel-cobalt oxide is compounded by mixing cobalt carbonate and nickel carbonate with a lithium carbonate, and calcinating at 900 degrees C.

[0008] Moreover, the approach of mixing cobalt, the hydroxide of nickel, and an oxide with a lithium is reported by JP,63-299056,A.

[0009] Coprecipitation of nickel ion and the cobalt ion is carried out as a carbonate out of the water solution which furthermore contains JP,1-294364,A nickel ion and cobalt ion, and the example which was mixed with the lithium carbonate after that and compounded the lithium compound nickel-cobalt oxide is reported.

[0010] However, in the nonaqueous electrolyte rechargeable battery which used for positive active material LiNiO₂ reported until now and LiNi_yCo_{1-y}O₂, there was a problem on which the cell engine performance deteriorates remarkably by performing the elevated-temperature retention test in the charge condition of a cell. As a result of this invention persons' repeating examination enough, it turned out that the thing of the following [degradation / such / property] is the cause.

[0011] As a result of disassembling the cell which carried out preservation degradation and performing the gas analysis within a cell case, and the analysis of electrolytic solution, the carbon-dioxide-gas concentration in the opening within a case was rising remarkably, and the decomposition product of an organic solvent was detected out of the electrolytic solution. Oxidative degradation of the organic solvent which is the electrolytic solution was carried out from these results, the decomposition product covered the active material front face, and degrading the property of a cell became whether to be **.

[0012] furthermore, BJH [distribution / the positive active material before and behind preservation / pore] using nitrogen adsorption -- as a result of performing detailed examination using law, that the decomposition product of an organic solvent has covered especially alternatively to the pore which has a pore radius 30A or less among pores 200A or less in positive active material became whether to be **.

[0013] Since the molecular size of the annular carbonate (for example, ethylene carbonate or propylene carbonate: describe it as Following EC and PC) used for an organic solvent and chain-like carbonate (for example, it is described as DMC and EMC below dimethyl carbonate and ethyl methyl carbonate:, respectively) was about 5-20A, this was considered that oxidative degradation is alternatively carried out in the pore which has comparable magnitude especially.

[0014] The purpose of this invention is offering the nonaqueous electrolyte rechargeable battery which aims at solution of the trouble about the above-mentioned conventional positive electrode, and was excellent in the elevated-temperature preservation property in a charge condition using better positive active material and a positive-electrode plate, holding high capacity and a good cycle property.

[0015]

[Means for Solving the Problem] In order to solve such a problem, we as a lithium compound nickel oxide used for the positive-electrode plate for nonaqueous electrolyte cells Chemical formula $\text{Li}_{x}\text{Ni}_{y}\text{M}_{1-y}\text{O}_2$ (it Co(es) x:1.10>=x>=0.98 and M) They are any one or more kinds of Mn, Cr, Fe, Mg, and aluminum, and the compound expressed with y:0.95>=y>=0.7. It is the particle to which primary particles 2 micrometers or less gathered, and the whole space product which it is made for the spatial body product which has a pore radius 30A or less to become 10% or less to the whole space volume, and has a pore radius 30A or less carries out to below 0.002cm³/g.

[0016] The electric conduction material which becomes this lithium compound nickel oxide from carbon material, a binder, and these are supported, and the positive-electrode plate for monotonous and constituted nonaqueous electrolyte cells which gives conductivity is used.

[0017] Furthermore, the positive-electrode plate which uses a lithium compound nickel oxide as a principal component and the negative-electrode plate which makes a subject electrochemically the carbon material or oxide of a lithium in which occlusion release reaction is possible, In the nonaqueous electrolyte cell which serves as the organic electrolytic solution, and a separator and the case which inserts these generation-of-electrical-energy elements from the obturation plate equipped with the relief valve before initial charge and discharge said lithium compound nickel oxide Chemical formula $\text{Li}_{x}\text{Ni}_{y}\text{M}_{1-y}\text{O}_2$ (it Co(es) x:1.10>=x>=0.98 and M) It is expressed with any one or more kinds of Mn, Cr, Fe, Mg, and aluminum, and y:0.95>=y>=0.7. It is the particle to which primary particles 2 micrometers or less gathered, and the spatial body product which has a pore radius 30A or less is 10% or less to the whole space volume. And the lithium compound nickel oxide which the whole space product which has a pore radius 30A or less made below 0.002cm³/g, The electric conduction material which consists of carbon material, a binder, and these are supported, and the amount of nonaqueous electrolyte per cell capacity 1Ah is set to 3/Ah 3.0-6.0cm using the positive-electrode plate for monotonous and constituted nonaqueous electrolyte cells which gives conductivity.

[0018] furthermore, a positive-electrode plate -- a weight ratio -- lithium compound nickel oxide:electric conduction material -- it is desirable for :binder to be 100:1-10:2-8.

[0019] said plate -- more than aluminum 95% -- it is desirable that it is the foil to contain. that a lithium compound nickel oxide is spherical or an ellipse -- a spherical thing is desirable.

[0020] As for a lithium compound nickel oxide, it is desirable for the BET specific surface area measured by nitrogen gas adsorption to be 0.15-0.3m²/g.

[0021] Mean particle diameter is 10-16 micrometers, and, as for a lithium compound nickel oxide, it is desirable for tap density to be three or more 2.0 g/cm.

[0022] Such a lithium compound nickel oxide can mix with lithium salt the different-species element dissolution nickel hydroxide expressed with chemical formula $\text{Ni}_{y}\text{M}_{1-y}(\text{OH})_2$ (M is any one or more kinds of Co, Mn, Cr, Fe, Mg, and aluminum, and y:0.95>=y>=0.7), and can obtain it by grinding and classifying the massive object

obtained by calcinating in a 700-900-degree C temperature requirement.

[0023] As for a lithium compound nickel oxide, eating into said plate is desirable. The solvent of nonaqueous electrolyte has large viscosity, and since the electrolytic solution does not permeate active material pore, it is [ethylene carbonate or a propylene carbonate independent] desirable [a solvent] that it is the mixed solvent which added chain-like carbonate with at least 20% or more of low viscosity, chain-like ester, or both at the rate of a volume ratio to these.

[0024] Moreover, as for chain-like carbonate, it is desirable that they are dimethyl carbonate or ethyl methyl carbonate, and, as for chain-like ester, it is desirable that it is methyl propionate.

[0025] As for a separator, it is desirable that it is a fine porosity film made from an organic giant molecule, and it is most desirable to consist of polyethylene, polypropylene, or an object that combined both.

[0026]

[Embodiment of the Invention] When the positive active material by this invention is used, the spatial body product which has a pore radius 30A or less considers as 10% or less to the whole space volume, the oxidative degradation of the organic solvent at the time of elevated-temperature preservation in the charge condition is further enabled to control remarkably, when the whole space product which has a pore radius 30A or less carries out to below 0.002cm³/g, and a good discharge property is acquired after preservation. Furthermore, since it is the particle to which primary particles 2 micrometers or less gathered and invasion to the interior of an active material particle of nonaqueous electrolyte becomes easy, the utilization factor of the active material at the time of charge and discharge improves.

[0027] moreover -- as a positive-electrode plate -- a weight ratio -- lithium compound nickel oxide:electric conduction material -- while being filled up with as many active materials in a cell as possible by constituting so that :binder may be 100:1-10:2-8, it becomes possible to fully hold the supply path of the electrolytic solution. Consequently, the positive-electrode plate which excelled [high capacity] in the charge-and-discharge property can be offered.

[0028] The positive-electrode plate of this invention, and the negative-electrode plate which makes a subject electrochemically the carbon material or oxide of a lithium in which occlusion release reaction is possible, In the nonaqueous electrolyte cell which serves as the organic electrolytic solution, and a separator and the case which inserts these generation-of-electrical-energy elements from the obturation plate equipped with the relief valve The nonaqueous electrolyte rechargeable battery which has the charge-and-discharge property which can distribute nonaqueous electrolyte appropriately in a positive electrode, a negative electrode, and a separator, and was excellent in setting the amount of nonaqueous electrolyte per cell capacity 1Ah to 3/Ah 3.0-6.0cm will be obtained.

[0029] Such effectiveness is not acquired only by limiting the configuration of an active material, and mean particle diameter like JP,6-267539,A, JP,1-304664,A, and JP,6-243897,A.

[0030] (Example 1) This invention is hereafter explained in accordance with a concrete example with a drawing.

[0031] Drawing of longitudinal section of the cylinder system cell used for drawing 1 by this example 1 is shown. The cell case into which 1 processed the stainless steel plate of organic-proof electrolytic-solution nature in drawing 1, the obturation plate with which 2 prepared the relief valve, and 3 show insulating packing. 4 is a group of electrode, and the positive-electrode plate 5 and the negative-electrode plate 6 are wound in the shape of a multiple-times swirl through a separator 7, and it is contained in the case. And from the above-mentioned positive-electrode plate 5, positive-electrode aluminum lead 5a is pulled out, and it connects with the obturation plate 2, and from the negative-electrode plate 6, negative-electrode nickel lead 6a is pulled out, and it connects with the pars basilaris ossis occipitalis of the cell case 1. 8 is prepared in the vertical section of a group of electrode 4 with the insulating ring, respectively.

[0032] Hereafter, the negative-electrode plate 6, the electrolytic solution, etc. are explained in detail. The negative-electrode plate 6 mixed the styrene-butadiene-rubber system binder in the graphite 100 weight section, and the carboxymethyl-cellulose water solution was made to suspend it in it, and it was made into the shape of a paste at it. And the front face of copper foil with a thickness of 0.015mm was plastered with this paste, and it rolled out to 0.2mm after desiccation, it started in width of face of 37mm, and magnitude with a die length of 300mm, and considered as the negative-electrode plate.

[0033] Hereafter, the synthesis method of positive active material is explained in detail. The sodium-hydroxide solution was added having introduced the nickel-sulfate solution and the cobalt sulfate solution in the container, and agitating them enough with constant flow, using a nickel sulfate, cobalt sulfate, and a sodium-hydroxide solution.

[0034] The nickel-cobalt compound hydroxide with various mean diameters was obtained by changing the addition of a sodium hydroxide.

[0035] The nickel-cobalt compound hydroxide which rinses generated sediment, dries and has various mean diameters was obtained.

[0036] All the chemical composition of the obtained nickel-cobalt compound hydroxide was nickel0.85Co0.15(OH)₂.

[0037] As a result of measuring mean particle diameter by laser diffraction, mean particle diameter was 5, 10, 13, and 16 or 18 micrometers, respectively.

[0038] The obtained nickel-cobalt compound hydroxide was mixed so that the mole ratio of a lithium hydroxide, Li, and nickel+Co might be set to 1.04:1, under the oxidizing atmosphere, it calcinated at 800 degrees C for 10 hours, and LiNi0.85Co 0.15O₂ (No.1-5) was compounded.

[0039] The obtained massive object was ground and classified and it considered as the active material for cells. It was checked that the compounded lithium compound nickel-cobalt oxide is a spherical aggregated particle to which many minute particles 2 micrometers or less come to gather by SEM observation.

[0040] The physical properties of the obtained lithium compound nickel-cobalt oxide are shown in (Table 1).

[0041]

[Table 1]

No.	平均粒径 (μ m)	タップ密度 (g/cm ³)	比表面積 (m ² /g)	空間体積比 (%)	30 Å 以下の 空間総体積 (cm ³ /g)	全細孔の総 体積 (cm ³ /g) 10 ~ 200 Å
1	4.76	1.95	0.99	5.2	0.00031	0.00596
2	10.02	2.35	0.30	6.5	0.00028	0.00432
3	13.20	2.73	0.23	8.3	0.00026	0.00311
4	16.43	2.95	0.15	9.1	0.00025	0.00274
5	18.89	3.06	0.11	12.1	0.00018	0.00149

[0042] The spatial body product ratio of (Table 1) is the rate of 30 or less spatial body product [as opposed to the whole space volume of 10-200 in a pore radius].

[0043] Moreover, as a result of analyzing Co contained by atomic absorption spectrophotometry in the lithium compound nickel oxide of 1-5, it checked containing Co by the mole ratio of 85:15 to nickel.

[0044] Moreover, although the inclination which becomes smaller than the ratio at the time of mixing was accepted in order that some Li might carry out the fly off of the mole ratio of Li and nickel+Co at the time of baking, it was checked that there is almost no difference in physical properties or a cell property 1.10:1 to 0.98:1.

[0045] In addition, it is thought that measurement is difficult and the space which has pore 10Å or less in fact exists by the approach according [pore distribution of 10Å or less] to nitrogen gas adsorption.

[0046] In addition, mean particle diameter was measured by the laser method, and made the value equivalent to 50% of accumulation mean particle diameter. Moreover, specific surface area was measured with the BET adsorption method which used nitrogen. Tap density filled up the 20 cc measuring cylinder (weight Ag) with the nickel-cobalt hydroxide, measured the volume Dcc of a weight (Bg) nickel-cobalt hydroxide after 200 times tapping, and asked for it by the degree type. [of a measuring cylinder]

[0047] Tap density (g/cc) = the manufacturing method of a positive-electrode plate is explained (B-A) / after D.

[0048] A positive-electrode plate mixes the acetylene black 3 weight section and the fluororesin system binder 5 weight section in the powder 100 weight section of LiNi0.85Co 0.15O₂ which is positive active material first, and N-methyl pyrrolidone solution is made to suspend it in it, and it is made into the shape of a paste at it. Both sides of aluminum foil with a thickness of 0.020mm were plastered with this paste, and it rolled out to 0.130mm after desiccation, it started in width of face of 35mm, and magnitude with a die length of 270mm, and considered as the positive-electrode plate 5. The paste was made into the coverage of each lot identitas.

[0049] An active material eating into aluminum core material mechanically, and being held was checked from the cross section of a positive-electrode plate, and surface observation of aluminum charge collector.

[0050] The film made from fine porosity made from polyethylene was used for the separator. And the positive-electrode plate and the negative-electrode plate were contained in the cell case with winding, a diameter [of 13.8mm], and a height of 50mm to the curled form through the separator.

[0051] After pouring into a group of electrode 4 three times 2.7cm using what was dissolved in the isochore product mixed solvent of EC and EMC at a rate of Roku phosphorus fluoride acid lithium 1 mol / 1, seal opening of the cell was carried out and it considered as the trial cell at the electrolytic solution. (bottom [the amount of electrolytic solutions per cell capacity 1Ah is equivalent to 3/Ah 4.5cm])

It examined under the following conditions using these cells.

[0052] After charging to 4.2V by 120mA under a 20-degree C environment, a pause is performed for 1 hour and it discharges to 3V by 120mA similarly after that. Charge and discharge were repeated 3 times by this approach, and 3rd discharge capacity was made into initial capacity.

[0053] Moreover, the utilization factor (mAh/g) of an active material was computed by breaking initial capacity by weight of the lithium compound nickel oxide contained in a cell.

[0054] Furthermore, after charging to 4.2V by 120mA, the cell was saved for 20 days under the 60-degree C environment, and after preservation repeated charge and discharge 3 times on the same charge-and-discharge conditions as the first stage, made 3rd discharge capacity the capacity after preservation, and computed the recovery factor after preservation using the degree type.

[0055]

[Equation 1]

$$\text{保存後回復率} = \frac{\text{保存後容量}}{\text{初期容量}} \times 100 \quad (\%)$$

[0056] The result of having investigated the utilization factor of the active material of the cell which used the lithium compound nickel oxide of 1-5, and the recovery factor after preservation is shown in (Table 2).

[0057]

[Table 2]

No.	初期容量 (mAh)	活物質利用率 (mAh/g)	保存後回復率 (%)	最大容量 (mAh)
1	610	177	91	625
2	607	173	86	651
3	609	175	84	666
4	612	175	79	688
5	605	173	62	672

[0058] The maximum capacity of (Table 2) is the capacity of the cell using the plate of the same size which applied the paste so that the pack density of a plate might become max. Maximum capacity is so large that restoration nature is high.

[0059] Although each utilization factor of the lithium compound nickel oxide of No.1-5 showed 170 or more mAh/g and the good property was acquired so that clearly from (Table 2), with the recovery factor after preservation, the lithium compound nickel oxide of No.5 showed 70% or less of very low value. Since the spatial body product ratio 30A or less with the high resolution of the organic solvent in the electrolytic solution was as large as 12% or more, the decomposition product covered the active material front face with disassembly of an organic solvent, and the active material of No.5 was considered to be the object with which the recovery factor after preservation fell in order to check an exchange of the electrolytic solution in the case of charge and discharge, and the electron between active materials. Moreover, the active material of maximum capacity of No.1 is low. The active material of No.1 was considered with since restoration nature is [tap density] low at less than three 2.0 g/cm.

[0060] That a lithium compound nickel oxide shows the preservation property which was excellent when the whole space product to which the spatial body product which has a pore radius 30A or less is 10% or less, and has a pore radius 30A or less to the whole space volume was below 0.002cm³/g became whether to be ** from the above result.

[0061] The lithium compound nickel oxide with such a property can mix with lithium salt the different-species

element dissolution nickel hydroxide expressed with chemical formula nickel $0.85\text{Co}0.15(\text{OH})_2$, and can obtain it by grinding and classifying the massive object obtained by calcinating at 800 degrees C.

[0062] In addition, a BET specific surface area and the spatial body product of pore have correlation in a spatial body product ratio, and, as for specific surface area and a spatial body product, it is desirable respectively to control in No.2 of this invention and the range of $0.15\text{-}3\text{m}^3/\text{g}$ and $0.0015\text{-}0.06\text{cm}^3/\text{g}$ shown in 3 and 4.

Furthermore, while the tap density of a lithium compound nickel oxide and mean particle diameter also have a spatial body product ratio and correlation, since it has big effect on the restoration nature to an electrode, it is important. A mean diameter is small like No.1, when tap density is small, it falls, the pack density, i.e., the capacity consistency, to an electrode of a lithium compound nickel oxide, and a substantial cell capacity falls.

[0063] Therefore, it is desirable for mean particle diameter to be 10-16 micrometers, and for tap density to be the range of $2.0\text{-}3.0\text{ g/cm}^3$.

[0064] (Example 2) As the 2nd example, the nickel-cobalt compound hydroxide of 13 micrometers of mean diameters used in the example 1 was mixed so that the mole ratio of a lithium hydroxide, Li, and nickel+Co might be set to 1.04:1, under the oxidizing atmosphere, calcinated, respectively at 600 degrees C, 700 degrees C, 900 degrees C, and 1000 degrees C, and the lithium compound nickel oxides 6, 7, 8, and 9 were compounded, and also the cell was created by the same approach as an example 1, and the retention test was performed.

[0065] The physical properties of the obtained lithium compound nickel-cobalt oxide are shown in (Table 3).

[0066]

[Table 3]

No.	合成温度 (°C)	空間体積比 (%)	30Å以下の空間 総体積 (cm ³ /g)	全細孔の総体積 (cm ³ /g) 10~200Å
6	600	4.1	0.00040	0.00987
7	700	4.5	0.00039	0.00868
8	900	4.3	0.00028	0.00651
9	1000	3.6	0.00022	0.00619

[0067] The result of having investigated the utilization factor of the active material of the cell which used the lithium compound nickel oxide of 6-9, and the recovery factor after preservation is shown in (Table 4).

[0068]

[Table 4]

No.	初期容量 (mAh)	活物質利用率 (mAh/g)	保存後回復率 (%)
6	458	131	87
7	609	174	92
8	573	163	91
9	428	122	86

[0069] No. compounded at 600 degrees C and 1000 degrees C although, as for the preservation property, each of lithium compound nickel oxides of No.6-9 showed 80% or more of good property about the recovery factor after preservation since a spatial body product ratio 30A or less and the whole space product were small so that clearly from (Table 3) and (Table 4) -- about 6 and 9, the utilization factor of an active material is very small with 131 and 122 mAh/g, respectively. As a result of investigating according to an X diffraction, in No.6 compounded at 600 degrees C, each full width at half maximum was large, and it was checked that the crystal is not fully growing.

Moreover, in No.9 compounded at 1000 degrees C, although crystal growth was fully progressing, the peak which does not belong hexagonal appeared. Investigation showed that this showed the rock salt structure to which nickel and Co fell to the lithium site. For this reason, the lithiums in which charge and discharge are possible decreased in number, and it was thought that it led to the capacity fall. As for the lithium compound nickel oxide of this invention, it is desirable to compound in a 700-900-degree C temperature requirement as mentioned above.

[0070] (Example 3) chemical formula Ni $y\text{Co}_{1-y}\text{(OH)}$ which has the mean particle diameter of 5 micrometers by

changing the addition of cobalt sulfate in the process which generates a nickel-cobalt compound hydroxide like an example 1 as the 3rd example -- two -- setting -- $y=0, 0.95, 0.9, 0.8, \text{ and } 0$. -- the nickel-cobalt compound hydroxide with the presentation of 7 and 0.6 was compounded.

[0071] The obtained nickel-cobalt compound hydroxide is mixed so that the mole ratio of a lithium hydroxide, Li, and nickel+Co may be set to 1.04:1. Under an oxidizing atmosphere, calcinate at 800 degrees C for 10 hours, and $\text{L}_x\text{N}_y\text{M}_1-y\text{O}_2$ ($7.0 < x < 1.10 >= x >= 0.98, y=0, 0.95, 0.9, 0.8, 0.6$) is compounded. the obtained massive object -- grinding -- classifying -- the object for cells -- it was referred to as active material No.10, and 11, 12, 13, 14 and 15.

[0072] The physical properties of the obtained lithium compound nickel-cobalt oxide are shown in (Table 5).

[0073]

[Table 5]

No.	平均粒径 ($\mu\text{ m}$)	y 値 ($\text{LiNi}_y\text{Co}_{1-y}\text{O}_2$)	空間体 積比 (%)	30 Å 以下の 空間総体積 (cm^3/g)	全細孔の総体積 (cm^3/g) 10~200 Å
10	12.67	1.00	4.4	0.00027	0.00610
11	13.33	0.95	6.2	0.00035	0.00566
12	13.61	0.90	3.4	0.00023	0.00682
13	12.24	0.80	3.5	0.00035	0.01003
14	13.11	0.70	4.4	0.00034	0.00766
15	12.57	0.60	5.1	0.00028	0.00553

[0074] The result of having investigated the utilization factor of the active material of the cell which used the lithium compound nickel oxide of 10-15, and the recovery factor after preservation is shown in (Table 6).

[0075]

[Table 6]

No.	初期容量 (mAh)	活物質利用率 (mAh/g)	保存後回復率 (%)
10	501	143	88
11	588	168	91
12	608	174	89
13	602	172	83
14	591	169	82
15	508	145	67

[0076] Although the preservation property showed 80% or more of good property about the recovery factor after preservation since a spatial body product ratio 30A or less and the whole space product were small, each of lithium compound nickel oxides of No.10-14, so that clearly from (Table 5) and (Table 6) In No.15 by which Co dissolved 40%, although a spatial body product ratio 30A or less and the whole space product are small, it turns out that the recovery factor after preservation is bad. As a result of investigating by X-ray diffraction, by the sample of No.15, the peak of LiCoO_2 and Co_3O_4 was accepted. From this, when the amount of Co dissolution exceeded 30%, the dissolution element did not dissolve completely, but it existed by LiCoO_2 and Co_3O_4 partially, especially Co_3O_4 acted as a decomposition catalyst of the electrolytic solution, and it was thought that the recovery factor after preservation was reduced. Moreover, in No.10 which are not dissolving Co, the utilization factor of an active material is small with 143 mAh/g. This had large polarization at the time of charge, and since the oxidative degradation electrical potential difference of the electrolytic solution was reached before being able to take out Li enough, it turned out that a large capacity expected is not obtained. LiNiO_2 is followed on it being reported in connection with the charge and discharge of a cell that the lattice constant changes (W. Li, J.N.Reimers and J.R.Dahn, Solid State Ionics, 67,123 (1993)), and being desorbed from Li, and Monoclinic and changing to the 2nd Hexagonal and the 3rd Hexagonal further are reported for the crystal phase from Hexagonal. It was considered the cause by which such crystal phase change became large [polarization]. By dissolving Co to some nickel, change

of such a crystal phase is eased remarkably. Since it was strong compared with nickel and the bonding strength with the oxygen of Co stabilized this more, the crystal structure is considered.

[0077] As for the amount of dissolution elements in $\text{Li}_{x}\text{Ni}_{y}\text{Ml}_{1-y}\text{O}_2$ ($x:1.10>=x>=0.98$) from these results, it is desirable that it is $0.95>=y>=0.7$.

[0078] In the process which generates a nickel compound hydroxide like an example 1 as the 4th example, nickel ion concentration in the water solution of a nickel sulfate is received. (Example 4) So that the concentration of an addition metal ion may be set to 85:15 Mn, Cr, Fe, The sulfate of Mg and aluminum was added and the nickel-cobalt compound hydroxide with the presentation of chemical formula $\text{nickel}0.85\text{M}0.15(\text{OH})_2$ (M is Mn, Cr, Fe, Mg, or aluminum) which has the mean particle diameter of 13 micrometers by the same approach as an example 1 was compounded.

[0079] The obtained nickel compound hydroxide is mixed so that the mole ratio of a lithium hydroxide, Li, and nickel+M may be set to 1.04:1. the bottom of an oxidizing atmosphere -- setting -- 800 degrees C -- 10 hours -- calcinating -- $\text{Li}_{x}\text{Ni}_{0.85}\text{M}0.15\text{O}_2$ ($x:1.10>=x>=0.98$ and M -- Mn --) the massive object which compounded Cr, Fe, Mg, or aluminum and was obtained -- grinding -- classifying -- the object for cells -- it was referred to as active material No.16, and 17, 18, 19 and 20.

[0080] moreover -- three -- a component -- a system -- ***** -- a nickel sulfate -- a water solution -- inside -- nickel -- ion concentration -- receiving -- addition -- a metal ion -- concentration -- 80:15:5 -- becoming -- as -- cobalt sulfate -- magnesium sulfate -- a sulfate -- adding -- an example -- one -- being the same -- an approach -- five -- micrometer -- mean particle diameter -- having -- a chemical formula -- nickel -- 0.80 -- Co -- 0.15 -- Mg -- 0.05 -- (OH) -- two -- a presentation -- having -- nickel - cobalt - magnesium -- compound -- a hydroxide -- having compounded .

[0081] the massive object which mixed the obtained nickel-cobalt-magnesium compound hydroxide so that the mole ratio of a lithium hydroxide, Li, and nickel+Co+Mg might be set to 1.04:1, calcinated at 800 degrees C under the oxidizing atmosphere for 10 hours, compounded $\text{Li}_{x}\text{Ni}_{0.80}\text{Co}_{0.15}\text{Mg}_{0.05}\text{O}_2$ ($x:1.10>=x>=0.98$), and was obtained -- grinding -- classifying -- the object for cells -- it was referred to as active material No.21.

[0082] The physical properties of the obtained lithium compound nickel-cobalt oxide are shown in (Table 7).

[0083]

[Table 7]

No.	平均粒径 (μm)	固溶元素	空間体 積比 (%)	30Å以下の空間 総体積 (cm^3/g)	全細孔の総体積 (cm^3/g) 10~200Å
16	13.21	Mn	4.1	0.00027	0.00652
17	13.68	Cr	5.6	0.00031	0.00553
18	12.22	Fe	4.4	0.00033	0.00742
19	13.31	Mg	5.2	0.00035	0.00669
20	12.69	Al	3.4	0.00029	0.00851
21	12.44	Co15%,Mg5%	4.4	0.00031	0.00701

[0084] The result of having investigated the utilization factor of the active material of the cell which used the lithium compound nickel oxide of 16-21, and the recovery factor after preservation is shown in (Table 8).

[0085]

[Table 8]

No.	初期容量 (mAh)	活物質利用率 (mAh/g)	保存後回復率 (%)
16	602	172	90
17	599	171	88
18	610	174	91
19	601	171	90
20	604	173	89
21	608	174	92

[0086] Since a spatial body product ratio 30A or less and the whole space product were small, as for the recovery factor after preservation, each of lithium compound nickel oxides of No.16-21 showed 80% or more of good property, so that clearly from (Table 7) and (Table 8).

[0087] Moreover, cell capacity is also understood that the large value is shown and the preservation property as Mn, Cr, Fe, Mg, and ***** for aluminum that it is the same besides Co can realize a good cell. Moreover, when both Co and Mg are made to dissolve, the same property is acquired, and same effectiveness can be realized also in 3 component system.

[0088] As an example 5, the lithium compound nickel oxide of No.3 used in the example 1 is used as an active material. (Example 5) In the powder 100 weight section of No.3, acetylene black and a fluororesin system binder are mixed at a rate shown in (Table 9). The cell which made N-methyl pyrrolidone solution suspend, and it was made the shape of a paste, and also created the positive-electrode plate like the example 1, and was created using this positive-electrode plate was made into cell A-J, respectively.

[0089]

[Table 9]

No.	活物質重量比	導電材重量比	結合剤重量比
A	100	0.5	5.0
B	100	1.0	5.0
C	100	5.0	5.0
D	100	10.0	5.0
E	100	15.0	5.0
F	100	3.0	1.0
G	100	3.0	2.0
H	100	3.0	4.0
I	100	3.0	8.0
J	100	3.0	10.0

[0090] The result of having investigated the utilization factor of the active material of cell A-J and the recovery factor after preservation is shown in (Table 10).

[0091]

[Table 10]

No.	初期容量 (mAh)	活物質利用率 (mAh/g)	保存後回復率 (%)
A	489	140	70
B	582	166	85
C	599	171	91
D	604	173	88
E	522	149	81
F	301	86	42
G	572	163	82
H	607	173	84
I	593	169	90
J	531	152	92

[0092] The amount of electric conduction material has become [the utilization factor of an active material] very as small as 140 mAh/g by 0.5 and few cells A to the active material 100 weight section, and it turns out that the conductive network of an active material is inadequate so that clearly from (Table 10). Like cell B-D, although the active material utilization factor and the recovery factor after preservation showed the good value like [the amount of electric conduction material / in 1 - 10 weight section] the cell of No.3 of an example 1, by the cell E with as many amounts of electric conduction material as [15 weight sections], the active material utilization factor became small with 149 mAh/g conversely.

[0093] When Cell E was disassembled and the positive-electrode plate was observed, while electric conduction material was condensing partially, the part to which the active material is not partially damp in the electrolytic solution was accepted.

[0094] The active material which the floc of electric conduction material absorbs the electrolytic solution, and the electrolytic solution does not permeate the whole positive-electrode plate, and cannot be contributed to a charge-and-discharge reaction from this was generated, and it was thought that the active material utilization factor as a cell decreased as a result.

[0095] As for the above result to the amount of electric conduction material, it is desirable that it is 1 - 10 weight section to the active material 100 weight section.

[0096] Next, as a result of examining the amount of binders, the amount of binders has become [the utilization factor of an active material] very as small as 86 mAh/g by 1.0 and few cells F to the active material 100 weight section. When Cell F was disassembled and the positive-electrode plate was observed, the remarkable thing dropped out was accepted from aluminum foil whose active material is a charge collector. Since the amount of binders shows a property good in 2.0 or more cell G-I, it turns out that the amount of binders is required more than the 2.0 weight sections in order to hold an active material to stability structurally at a charge collector. Moreover, the inclination for the amount of binders to become [an active material utilization factor] conversely as small as 152 mAh/g by the cell J of the 10.0 weight sections was accepted. When this had too many charge-and-discharge reactions and binders which do not participate in electronic conduction, the active material front face was covered, in order that surface area effective in a charge-and-discharge reaction might decrease, polarization became large, and it was considered that the utilization factor decreased.

[0097] As for the above result to the amount of binders, it is desirable that it is 2 - 8 weight section to the active material 100 weight section.

[0098] As an example 6, the positive-electrode plate using the active material of No.3 used in the example 1 as an active material is used. (Example 6) As the electrolytic solution, seal opening of the cell was carried out and the amount poured in a group of electrode 4 using what was dissolved in the isochore product mixed solvent of EC and EMC at a rate (6 phosphorus-fluoride acid lithium 1mol/l.) was made into trial cell K-P, after pouring in three times 3.6 or 4.0cm, 1.6, 1.8, 2.5, 3.0, and.

[0099] The test result of cell K-P is shown in (Table 11).

[0100]

[Table 11]

No.	電解液量 (cm ³)	1Ah 当たりの 電解液量 (cm ² /Ah)	初期容量 (mAh)	活物質利用率 (mAh/g)	保存後回復率 (%)
K	1.6	2.80	571	163	83
L	1.8	2.98	604	173	90
M	2.5	4.19	596	170	92
N	3.0	4.98	602	172	89
O	3.6	5.99	601	171	91
P	4.0	6.55	603	172	—

[0101] By the cell K with few amounts of electrolytic solutions, the utilization factor of an active material is falling so that clearly from (Table 11). This is considered because the electrolytic solution has not spread round the active material front face enough. Moreover, by the cell P with many amounts of electrolytic solutions, there is much excessive electrolytic solution, in order to occupy the volume of voids in a cell, the internal pressure in a cell rises with the carbon dioxide gas which occurs at the time of elevated-temperature preservation in the charge condition, and actuation and a cell came to spill liquid [the relief valve].

[0102] The amount of electrolytic solutions with the cell more nearly optimal than the above result is 3/Ah 3.0-6.0cm per cell capacity 1Ah.

[0103] (Example 7) As an example 7, the positive-electrode plate using the active material of No.3 used in the example 1 as an active material was used, and trial cell Q-Z, and AA and AB were created using what was dissolved in the non-aqueous solvent of the volume ratio presentation shown as the electrolytic solution in (Table 12) at a rate (6 phosphorus-fluoride acid lithium 1mol/l.).

[0104]

[Table 12]

No.	EC	PC	EMC	DMC	MP
Q	100				
R		100			
S	80		20		
T	20		80		
U		80	20		
V		20	80		
W	50			50	
X	50				50
Y	20		70		10
Z	20			70	10
AA		20	70		10
AB		20		70	10

[0105] The test result of cell Q-Z, and AA and AB is shown in (Table 13).

[0106]

[Table 13]

No.	初期容量 (mAh)	活物質利用率 (mAh/g)	保存後回復率 (%)
Q	502	143	80
R	491	141	82
S	602	172	90
T	598	171	84
U	607	173	86
V	602	172	88
W	599	171	91
X	592	170	85
Y	604	173	86
Z	600	171	83
AA	606	173	90
AB	602	172	91

[0107] By the cells Q and R whose solvents of the electrolytic solution are EC or a PC independent solvent, the utilization factor of an active material is small with 150 or less mAh/g so that clearly from (Table 13). When such an independent solvent was used, this had the large viscosity of the electrolytic solution and was considered that a substantial specific surface area of an active material became small since the electrolytic solution was not filled by the pore in the active material which is the reaction place of a charge-and-discharge reaction, and polarization became large.

[0108] On the other hand, by cell S-Z which added at least 20% or more of chain-like ester (EMC, DMC) or chain-like ester (MP) at the rate of a volume ratio to ethylene carbonate or propylene carbonate, and AA and AB, each showed the good cell property.

[0109] As for the solvent of nonaqueous electrolyte, it is more desirable than the above result that it is the mixed solvent which added at least 20% or more of chain-like carbonate, chain-like ester, or both at the rate of a volume ratio to ethylene carbonate or propylene carbonate, and, as for dimethyl carbonate or ethyl methyl carbonate, and chain-like ester, it is [chain-like carbonate] desirable that it is methyl propionate.

[0110] (Example 1 of a comparison) As an example 1 of a comparison, the configuration of a particle compounded lithium compound nickel-cobalt oxide No.22 like the example 1 by making a massive nickel-cobalt compound hydroxide into a raw material. The chemical composition of the obtained nickel-cobalt multiple oxide was LiNi0.85Co 0.15O2.

[0111] The compounded lithium compound nickel-cobalt oxide was obtained as a massive particle whose mean particle diameter is 12 micrometers.

[0112] The property of the obtained lithium compound nickel-cobalt oxide is shown in (Table 14).

[0113]

Table 14]

No.	平均粒径 (μ m)	タップ密度 (g/cm ³)	比表面積 (m ² /g)	空間体積比 (%)	30 Å 以下の 空間総体積 (cm ³ /g)	全細孔の総 体積 (cm ³ /g) 10 ~ 200 Å
22	12	2.55	0.12	16.7	0.00016	0.00096

[0114] The lithium compound nickel-cobalt oxide of No.22 was used as positive active material, and also the cell was created like the example 1.

[0115] The result of having investigated the utilization factor of the active material of the cell which used the lithium compound nickel oxide of No.22, and the recovery factor after preservation is shown in (Table 15).

[0116]

[Table 15]

No.	初期容量 (mAh)	活物質利用率 (mAh/g)	保存後回復率 (%)
22	497	142	63

[0117] In order for a decomposition product to cover an active material front face with disassembly of an organic solvent since the spatial body product ratio 30A or less with the high resolution of the organic solvent in the electrolytic solution is as large as 16.7%, and to check an exchange of the electrolytic solution in the case of charge and discharge, and the electron between active materials, the active material of No.22 has a low recovery factor after preservation, so that clearly from (Table 14) and (Table 15). Moreover, since specific surface area is small especially since it is a massive particle, and polarization at the time of being charge and discharge is large, the utilization factor of an active material is also small. the particle to which primary particles 2 micrometers or less gathered also in the nickel compound hydroxide which serves as a raw material of a lithium compound nickel oxide from the above result not to mention a lithium compound nickel oxide -- it is -- spherical or an ellipse -- a spherical thing is desirable.

[0118] Although evaluated using the cylindrical cell in the above-mentioned example, the same effectiveness is acquired even if cell configurations, such as a square shape, differ.

[0119] Furthermore, although the graphite was used for the negative electrode in the above-mentioned example, in order that the effectiveness in this invention may act in a positive-electrode plate, the same effectiveness is acquired even if it uses other negative-electrode ingredients, such as an oxide of other amounts of carbonaceous material, a lithium metal, a lithium alloy, Fe2O3, WO2, and WO3 grade.

[0120] Moreover, although the 6 phosphorus-fluoride acid lithium was used as an electrolyte in the above-mentioned example, effectiveness with the same said also of 4 other lithium content salt, for example, lithium perchlorate, and lithium borate fluoride, a trifluoro methansulfonic acid lithium, and a 6 fluoride [arsenic acid] lithium was acquired.

[0121]

[Effect of the Invention] The nonaqueous electrolyte rechargeable battery the elevated-temperature preservation property in a charge condition excelled [rechargeable battery] in high capacity can be offered by creating the cell of a configuration of having optimized the amount of electrolytic solutions and the electrolytic-solution solvent kind further using the electric conduction material which optimized the addition, and the positive-electrode plate which added the binder using the lithium compound nickel oxide by this invention so that clearly from the above explanation.

[Translation done.]